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CZOCHRALSKI RUBY

Final Report

Period: January 1, 1967 - April 30, 1967

February 10, 1968

Contract No. Nonr-4132(00)

UNION CARBIDE

UNION CARBIDE CORPORATION
ELECTRONICS DIVISION

P. O. Box 24184
Indianapolis, Indiana 46224

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Contract Nonr-4132(00)
Program Code Number 3730
Authorization ARPA Order 306-62
Task Number NR017-710

CZOCHRALSKI RUBY

Final Report

Period: January 1, 1967 - - April 30, 1967

February 10, 1968

Principal Investigator:
F. R. Charvat

Work Performed by:
G. A. Keig
L. R Rothrock
J. M. J. Watts

Report Written by:
G. A. Keig

This research is a part of Project DEFENDER under the joint sponsorship of the Advanced Research Projects Agency, the Office of Naval Research and the Department of Defense.

Union Carbide Corporation
Electronics Division
Crystal Products R and D
P. O. Box 24181
Indianapolis, Indiana 46224

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I. INTRODUCTION

The object of this program has been to develop the Czochralski technique to yield large ruby crystals with optical quality suitable for use as solid state lasers. This development has required considerable effort both in the setting up of a suitable furnace of the required size and stability to grow the large crystals from, and in the fabrication of end faces with a sufficiently good finish to allow a satisfactory optical evaluation to be carried out.

Work has continued towards obtaining an improved understanding of the optical inhomogeneities in the ruby. Special emphasis has been placed on the core as this has been shown to introduce an undesirable energy distribution during lasing. Core-free rods have been fabricated from the large diameter boules and their behavior under both passive and active conditions compared to that of rods containing the core.

II. EXPERIMENTAL RESULTS

A. Growth of Large Diameter Ruby Boules

The majority of our growth effort during the final six months of the project has concentrated on the modification of the furnace construction and design in order to come up with the optimum conditions for reproducible growth.

As mentioned previously [(1)], the generator being used for the growth experiments had too low a power rating and problems arose because it was being operated close to its maximum rated output. In order to reduce these problems, a series of modifications were carried out on both the generator, and the furnace insulation and design; some measure of success was achieved with these modifications and a few crystals of good quality were grown and evaluated.

The crystal growth was able to proceed in a more satisfactory and reproducible fashion after a generator with an additional 5 KW output was installed and readied for use. The internal design of this generator was sufficiently different that some minor changes in coil design were required in order to achieve the most efficient coupling. However, the largest contribution to successful growth was brought about with the realized increase in available power and the increased stability and reliability through being able to operate the generator sufficiently below its rated maximum.

A problem in the quality control of large diameter ruby was brought about through the large physical dimensions of the "as-grown" boules, where a small change in the boule diameter was responsible for a relatively large change in growth rate at the solid-liquid interface. Thus careful attention had to be paid to the control system in order to keep the diameter fluctuations at a minimum and prevent the nucleation of micro-bubbles at the interface through corresponding changes in growth rate. This problem, which increases in severity with increased chromium concentration, has been solved successfully and boules can currently be grown with almost undetectable diameter changes along their entire length.

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(1) "Czochralski Ruby", Report No. SRCR-67-5 by G. A. Keig, O. H. Nestor, J. C. Smith, P. E. Otten, Union Carbide Corporation, Electronics Division, March 20, 1967 (Annual Technical Summary Report, Contract Nonr-4132(00).

Fabrication of flat and parallel ends on to these large diameter rods was also somewhat of a problem as our polishing technique relied on grinding down the rods on the diameter prior to finishing the ends. Because of the interest in knowing the optical quality across the entire diameter of the "as-grown" crystal, it was decided to develop a technique to place acceptable ends on to the raw boule. This has arrived at the point where good Schlieren photographs can be taken of the crystal, although further improvement is required in order to obtain the maximum amount of information from the Twyman-Green interferograms.

The size of the grown crystals has been limited by the size of the iridium crucible and the corresponding volume of the melt it can contain, and to grow crystals between 10 and 12 inches long has required a restriction on the diameter to slightly below 2 inches. The chromium oxide concentrations used for these growths have been 0.015 weight % and 0.05 weight % and a representative selection of crystals is shown in Figure 1. It has been possible, however, to extend the diameter to 2-1/4 inches, while restricting the length to slightly under 8 inches, and a crystal with these dimensions and containing 0.05 weight % chromium oxide is shown in Figure 2; this crystal could be turned down to produce a rod with a finished ground diameter in excess of 2 inches.

The largest crystal grown to date has a diameter of 2-1/4 inches, and a length restricted to 8 inches by the dimensions of the crucible; with a larger size crucible, this length could readily be extended to greater than 12 inches while retaining the same diameter. Only minor modifications in the furnace design would be required in order to accommodate the larger crucible necessary for the growth of crystals from which a finished rod with dimensions of 2-inch diameter and 13 inches long could be fabricated. All the other technical skills which have been developed would be used without any anticipated change.

The fabricated rod size given above satisfies the overall dimensions proposed as our ultimate goal at the completion of the project. However, our experiments to date show no indication that this represents the maximum possible size, and larger crystals could be grown if the need arose without an unreasonable amount of additional technical effort.

FIGURE 1. A representative selection of ruby boules with diameters of approximately 2 inches.

FIGURE 2. A ruby boule containing 0.05 weight % Cr_2O_3 and having a diameter just over 2-1/4 inches.

B. Optical Quality of the Large Diameter Boules

Large diameter ruby single crystals can be utilized in two different ways:

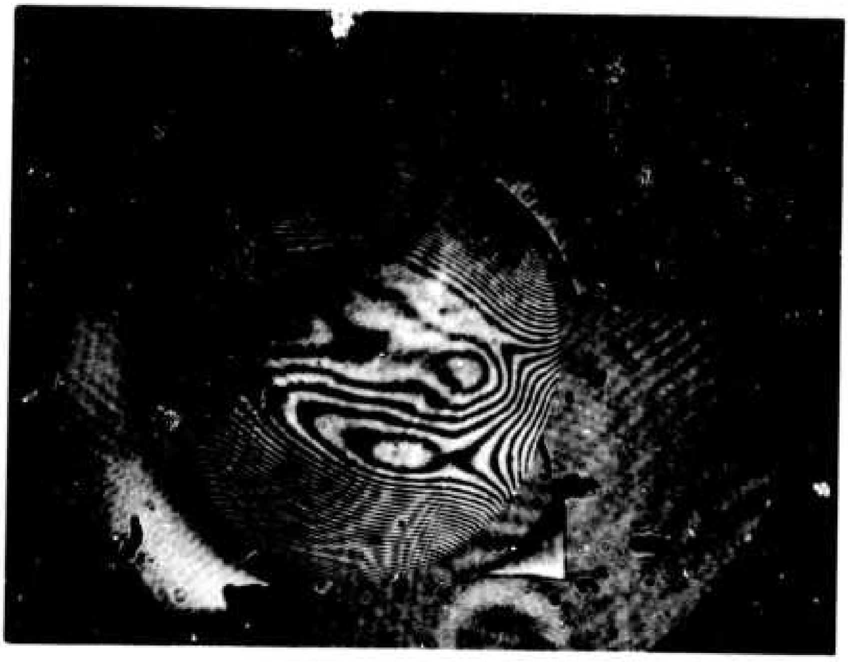
(a) to produce large diameter laser rods which will be used as amplifiers in a system designed to produce high power laser output;

(b) to obtain smaller diameter laser rods of superior optical quality; this can be achieved through fabricating the rods from sections of the large diameter boules.

Both of these applications require a boule with the optimum optical quality which makes it necessary to have as good an evaluation as possible of the grown material. This type of evaluation is best realized by taking either a Schlieren photograph, or an interferogram, using the Twyman-Green interferometer, down the length of the rod. The Twyman-Green interference method allows a measure of the average change in refractive index down the rod length (this change can come about through strain and/or variations in solute concentration), whereas the Schlieren technique is best suited to detecting small localized changes in refractive index (i. e., the core, which usually extends down the center of the rod).

Figure 3 shows the Twyman-Green interference pattern for a large diameter crystal with a nominal chromium oxide concentration of 0.015 weight %. Most of the fringes observed are due to the end faces not having the necessary degree of flatness and parallelism for obtaining meaningful interference patterns, and more information can be obtained by adjusting the mirrors to compensate for the deficiencies in end finish. This adjustment has the advantage of enabling simplified patterns for portions of the face to be obtained, which can be pieced together to form a composite pattern.

In order to yield a crystal which could readily be fabricated without further annealing, special precautions were taken in cooling the crystal by slowly withdrawing it from the melt through a system of ceramic baffles. This cool down procedure,



TWYMAN-GREEN

E Parallel to C

E

⟵⟶

SCHLIEREN

FIGURE 3. Optical evaluation of large diameter ruby containing 0.015 weight % Cr_2O_3.

although it achieved the purpose of producing a crystal with sufficient strength to withstand fabrication, also produced a somewhat complex stress pattern due to a temperature gradient and differential cooling along the length as well as across the diameter. The sample shown in Figure 3 has not been annealed and this factor is reflected in the Twyman-Green pattern.

A considerable amount of strain is concentrated in a narrow region adjacent to the outside surface of the crystal, presumably due to a very rapid temperature drop in this outside skin through radiative heat transfer during the initial stages of cooling. An improvement in the overall optical quality can be brought about by grinding down the outside of the crystal into the form of a rod with uniform diameter; this procedure also assists with the attainment of flat and parallel ends for inspection purposes. Figure 4 shows a fabricated ruby rod with diameter slightly in excess of 1-3/4 inches and containing 0.05 weight % chromium oxide, and Figure 5 the corresponding Twyman-Green and Schlieren photographs. The quality of this rod is good and could be improved further through annealing.

The extent of the core in ruby is a function of both the particular growth conditions used and the growth orientation [1], and based on our previous experience the seed orientation for all the growth experiments was chosen with the closest "r" plane normal between 10-15 degrees from the direction of pull. The orientation of the seed direction with respect to the normal to the closest "r" plane, and along the a and c direction are given in Table I for seeds used in the growth of large diameter rubies.

TABLE I

Seed No.	r_1	c	a
112	15	66-1/2	30
115	14	65	30
166	12	60-1/2	34

As indicated by the Schlieren photographs in Figures 3 and 5, this choice of orientation produces a boule with the core tightly confined down the center due to the formation of only one facet on the growth interface in the vicinity of the tip. This property is extremely useful as it produces large areas of the boule free from the inhomogeneity caused by the core and from which relatively large laser rods with superior optical quality can be fabricated.

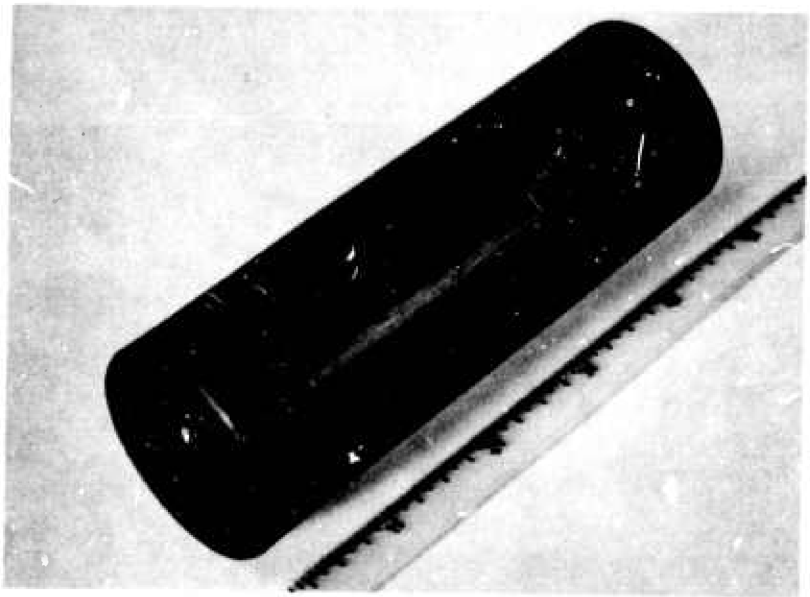
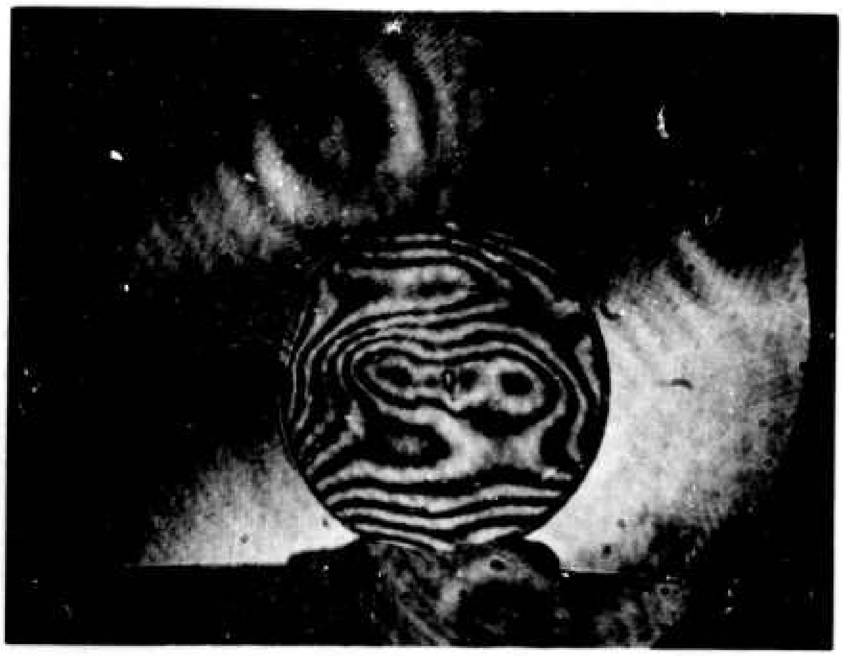


FIGURE 4. Ruby boule fabricated to a uniform diameter of 1-3/4 inches.



TWYMAN-GREEN

E Perpendicular to C

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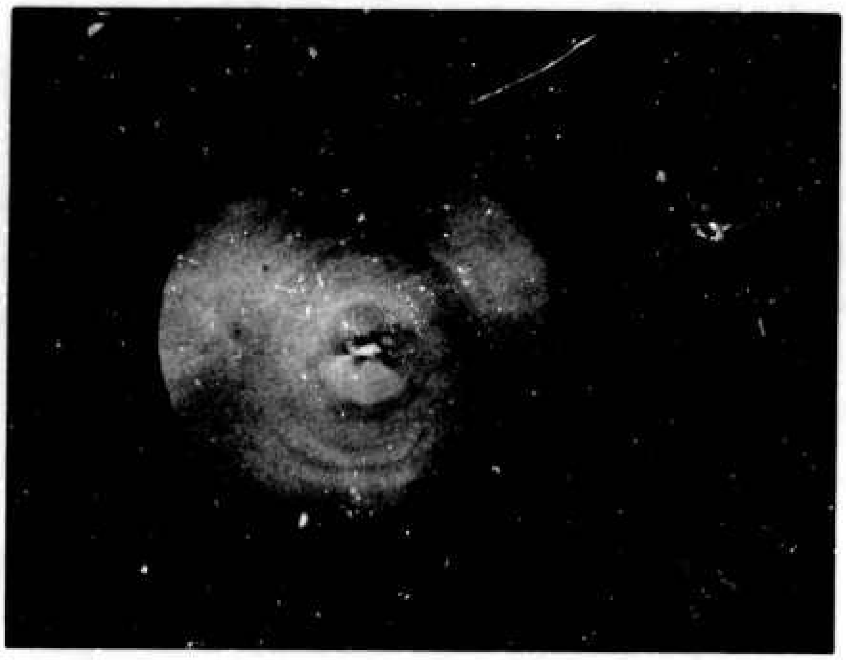

SCHLIEREN

FIGURE 5. Optical evaluation of large diameter fabricated rod.

Figure 6 shows a rod 3/4-inch diameter and 8-1/2 inches long which has been fabricated from a section of a large diameter boule with a nominal chromium oxide concentration of 0.015 weight percent. The Schlieren photograph shows the rod to be of good optical quality and free from any core structure down its length; the ends of this rod were not finished to laser tolerances and surface scratches can be seen on the photograph.

The improvement in optical quality brought about through the use of a high temperature anneal is demonstrated by the series of Twyman-Green patterns shown in Figure 7. The top photograph shows a pattern of the fabricated rod in its "as grown" state and indicates a relatively large number of fringes due to stresses introduced during growth and fabrication. The bottom photographs show how a high temperature anneal has been successful in relieving the residual stresses and reducing the number of fringes to one along an 8-inch rod length. The refinished ends of the annealed rod were checked and found to be flat to within 0.2 and 0.1 of the wavelength of a helium monochromatic source (5875 Å), and parallel to within 7 seconds of arc.

In addition to changes in refractive index down the length of the rod, optical inhomogeneities can also arise through the presence of small localized inclusions which act as scattering centers for the laser beam, and which are known to cause damage under high power Q-switched operation.

With the exception of two boules where the growth station was incorrectly set up, no inclusions have been detected in the large diameter rubies using the standard technique of shining a collimated beam of light into the crystal and observing the scattered radiation. However, because of the amount of absorption encountered with such a large diameter crystal, it was felt that the preliminary results could be misleading due to a light with too low an intensity being used. A further check with a light source of higher intensity on the large rods as well as smaller diameter rods fabricated from sections of the large rods confirmed our previous findings that within the limits of our experimental measurements, there were no visible inclusions.

In general, the optical quality of the large diameter boules is good and is comparable to unannealed boules grown with smaller dimensions on a production basis. Improvement in quality is realized by grinding off the highly stressed outer skin; annealing the crystals under the correct conditions would almost certainly bring about a further improvement.

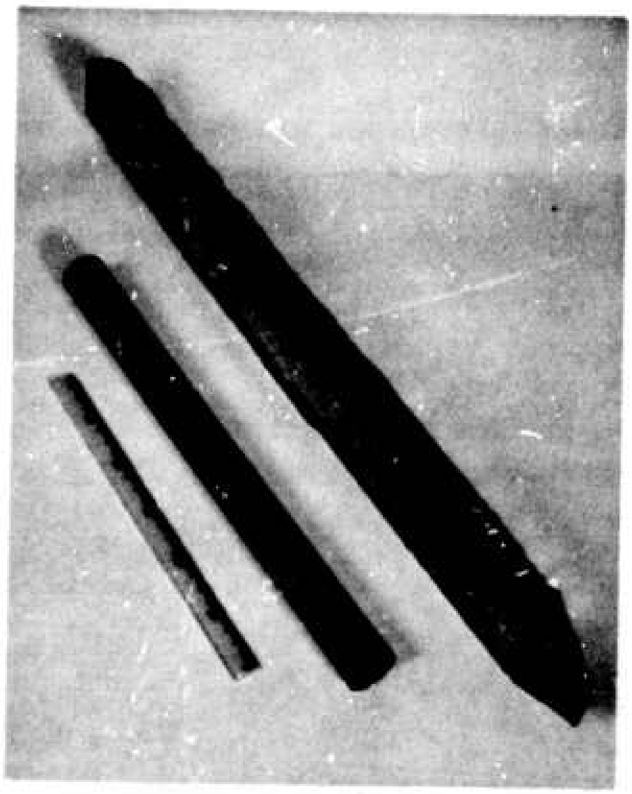

FIGURE 6. The 3/4-in. diameter rod fabricated from a section of a large diameter rod and an evaluation of its optical quality from a Schlieren photograph.

BEFORE ANNEALING

AFTER ANNEALING

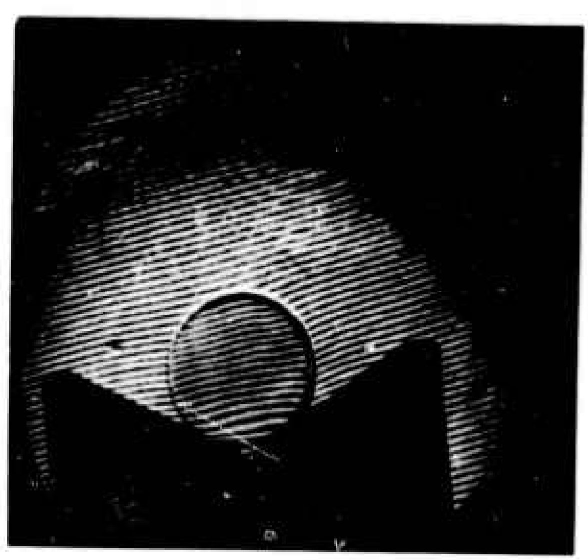

FIGURE 7. Twyman-Green photograph of fabricated 3/4-inch diameter x 8-1/2-inch long rod showing improvement in optical quality after a high temperature anneal.

1. Further Investigation of Core

As reported previously, the core in ruby represents a localized area of higher chromium concentration extending throughout the length of the crystal. This increase is brought about through the formation of "r" plane facets on the growth interface which have a different effective distribution coefficient than the average for the remainder of the growing interface.

The distribution of chromium across the core has been determined on polished sections of the boule using the optical absorption technique and a correlation made between the core, and the shape and size of the facet on the interface.[(1)] A further study has been carried out to determine the details of the growth facet using optical microscopy and the electron microprobe [(2)].

Figure 8A is a photograph of a facet at the tip of the growth interface in ruby and Figure 8B shows the microscopic detail of a portion of the facet close to tip; the remainder of the facet was planar and showed no further detail that could be observed with normal illumination. This structure is interesting and suggests the possibility that growth on the facet takes place by some form of step process. This is not entirely unexpected as the step represents an active site for the addition of atoms on a low energy plane, and the probability of atoms moving across the solid surface and remaining in the vicinity of the step is much higher than it would be for them to affix themselves on to the remainder of the planar surface.

Figure 8C and 8D show an electron image scan and x-ray image scan carried out in the vicinity of the growth steps (for the latter, the Cr K, α, line was used to determine the distribution of chromium). A definite increase in chromium concentration was observed in the vicinity of the growth steps and at the center of the elevated regions.

---

(2) The electron microprobe work was carried out at Union Carbide Mining and Metals Division, Technology Department, Niagara Falls, New York, under the direction of Dr. W. D. Forgeng.

A

B

C X250

D X250

FIGURE 8. The microscopic details and chromium distribution along a portion of the growth facet.

If we consider that growth is taking place by the step process, then the fastest growth direction is normal to the facet (r-plane) and determined by the rate at which atoms are adsorbed at the step. Burton, Prim, and Slichter [3] have given an effective distribution coefficient for solute during solidification as:

$$K_E = \frac{Ko}{Ko - (Ko - 1)\ \exp.\ \left(-\frac{R\,\delta}{D}\right)}$$

R = growth rate

D = diffusion coefficient of solute in the liquid

K_o = equilibrium distribution coefficient for the solute

δ = the distance from the growing interface, beyond which the fluid flow keeps the concentration uniformly equal.

For chromium solid solution in aluminum oxide, the equilibrium distribution coefficient has been calculated to be equal to 2.1 [4]. For an infinitely slow growth rate the effective distribution coefficient K_E tends towards Ko, whereas an increase in growth rate decreases the value of K_E towards a minimum value of unity. Because K_E decreases with growth rate more solute will be rejected across the growing interface in the vicinity of the step than across the remainder of the facet, and it is this increase in chromium concentration that is measured with the x-ray scan on the electron microprobe.

There are many unanswered questions concerning facet formation, localized growth in the vicinity of or across the facet, and the effect of these non-uniform growth conditions at the interface on the internal quality of ruby laser rods. The work carried out for this contract has made an initial step and should allow more meaningful and fruitful experiments to be planned for future effort along these lines.

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(3) J. A. Burton, R. C. Primm, and W. P. Slichter, J. Chem. Phys., 21, 1987 (1953).

(4) "Growth of Ruby Crystals by Pulling from the Melt", Linde Research Report No. 138, by M. N. Plooster, Union Carbide Corporation, Linde Division, Nov. 25, 1964.

2. The Effect of the Core on Lasing Performance

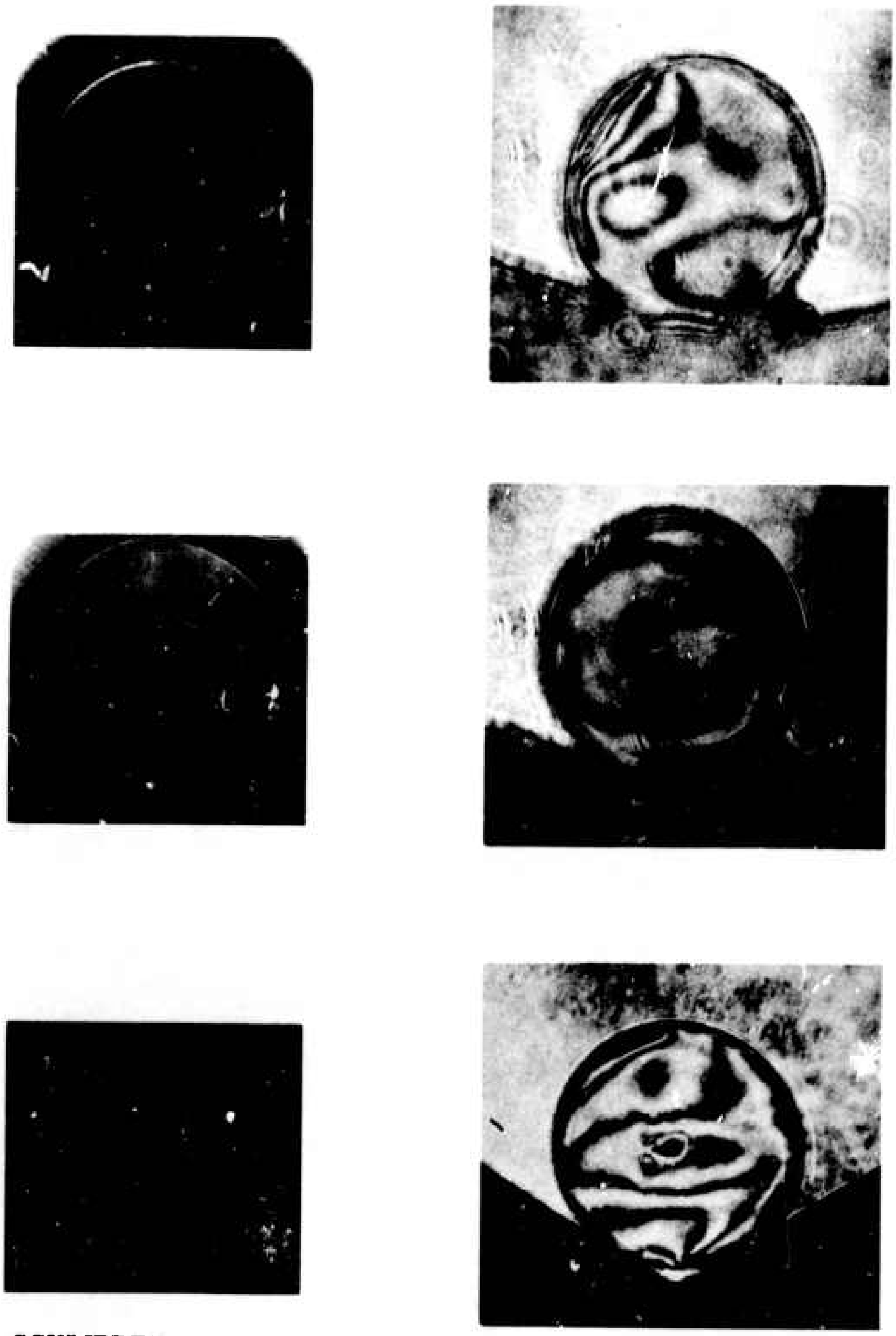
It has been demonstrated conclusively both at our laboratory and from outside sources, that for both Verneuil and Czochralski ruby laser rods the energy is not uniformly distributed across the lasing area. For Verneuil material, the main contribution comes from internal stresses and uneven chromium distribution, whereas for Czochralski material it is the core which proves the main source of optical inhomogeneity.

In order to fully understand the effect of the core on the lasing behavior of ruby rods a series of experiments have been conducted using two sets of rods. The first set contained the central core after fabrication, whereas the others were specially selected from areas of the large diameter boules to be free from this central defect. All the experiments carried out under active lasing conditions used a Korad K2 laser head. The ruby rods with dimensions 1/2-inch diameter and 6 inches long were tested under normal mode operation.

The program had two objectives: (1) to find a correlation between the passive and active tests and (2) see what improvements could be brought about in the uniformity of output energy through removal of the core.

Figure 9 shows the Twyman-Green and Schlieren photographs of three laser rods, ON 251 and ON 252 being core-free and ON 253 with the core remaining. Figure 10 shows active near field photographs for the three rods at energy outputs of 4 J and 20 J respectively.

As can be seen, a clear correlation exists between the passive Schlieren photographs showing the core and the near field lasing pattern. In general it is observed that the core tends to lase first with increasing input energy (this includes the case of both the central and satellite cores) and leads to an uneven and undesirable energy distribution. There is no obvious correlation between far field patterns obtained under passive and active conditions.



ON 251

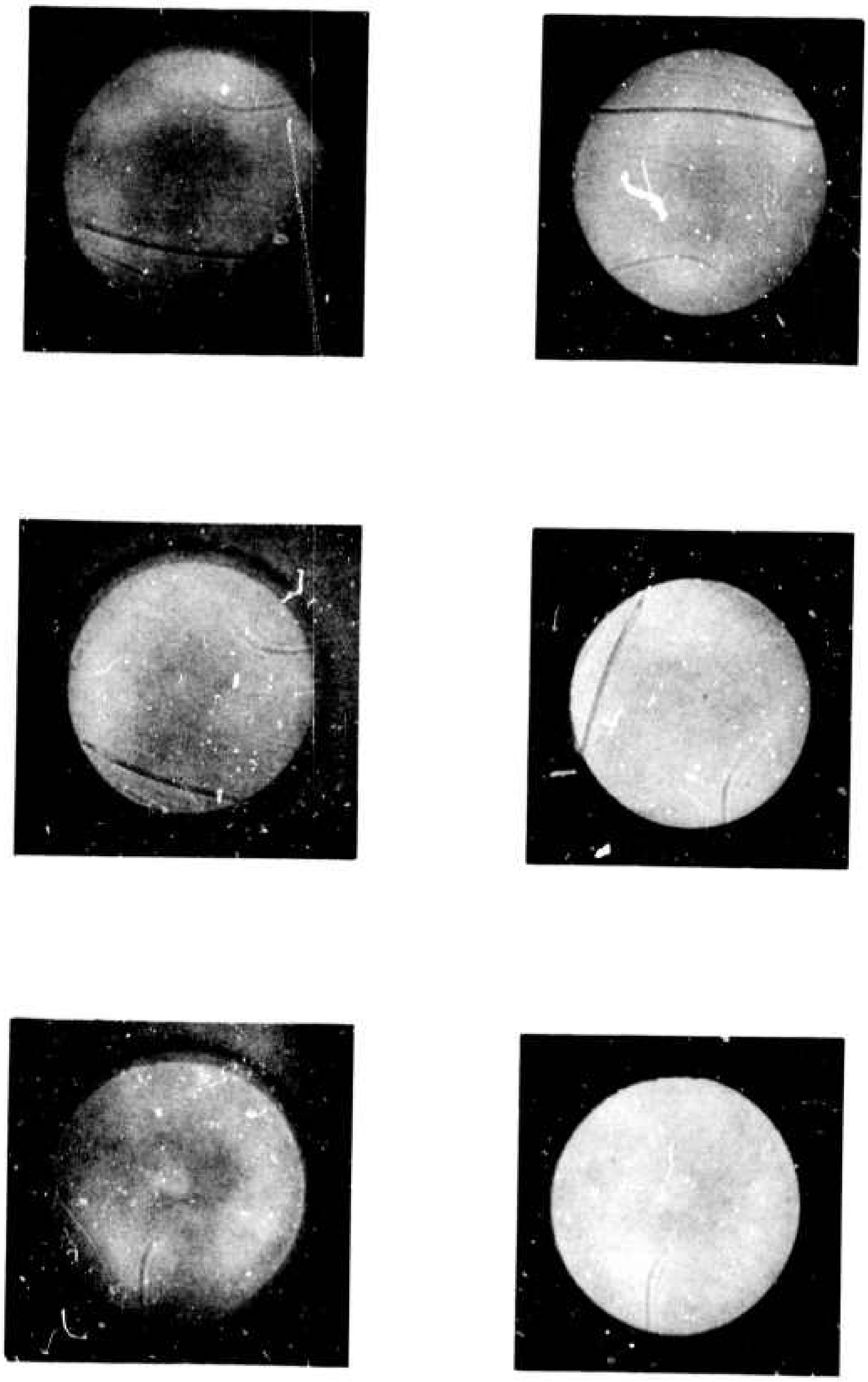
ON 252

ON 253

SCHLIEREN

TWYMAN-GREEN

FIGURE 9. Passive optical evaluation of rods used to determine the effect of the core on lasing behavior.



ON 251

ON 252

ON 253

4 Joules Output

20 Joules Output

FIGURE 10. Near field lasing pattern of the three test rods for different energy output.

Beam divergence measurements have been made under passive and active conditions and the results are shown in Figures 11 and 12. In each case, the fraction of the total energy contained in the included angle of the far field cone is given as a function of the conical angle. The passive beam divergence measurements do not show a big difference between the rods with and without the core due to the fact that the light makes only a single pass through the rod. A more pronounced and definite improvement is seen, however, when measurements are carried out under active lasing conditions where ruby acts as an oscillator and the laser light makes multiple passes along the length of the rod. In this way the inhomogeneity is magnified and the resulting lack of uniformity of the rod made more evident.

If a measure is made of the conical angle for 50% and 90% of the output energy contained in the same, there is an improvement of 26% and 40%, respectively, between the uncored and cored rods. These improvements are especially significant when the brightness is considered which is proportional to the square of the above.

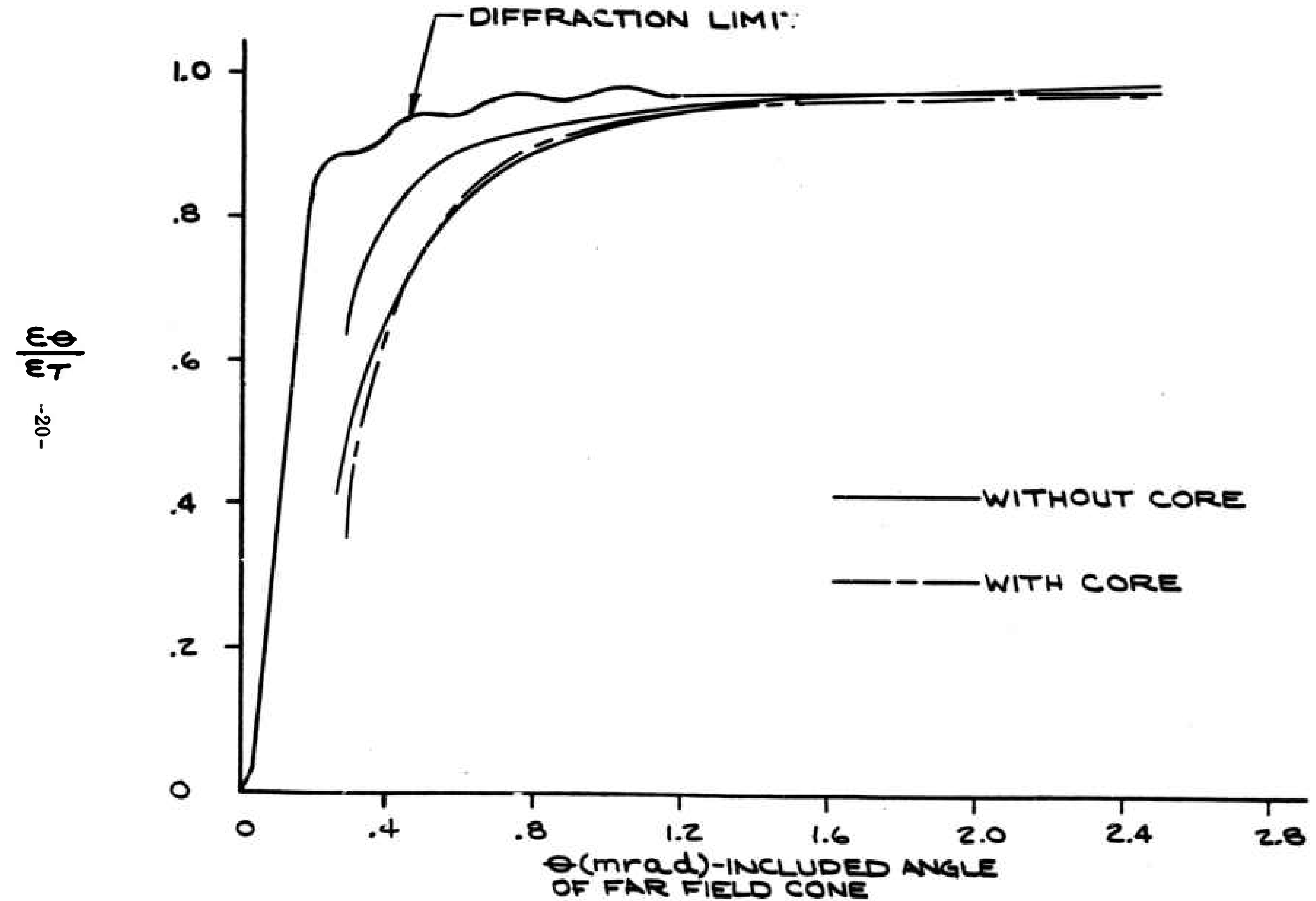


FIGURE 11 - BEAM DIVERGENCE RESULTS OBTAINED UNDER PASSIVE CONDITIONS

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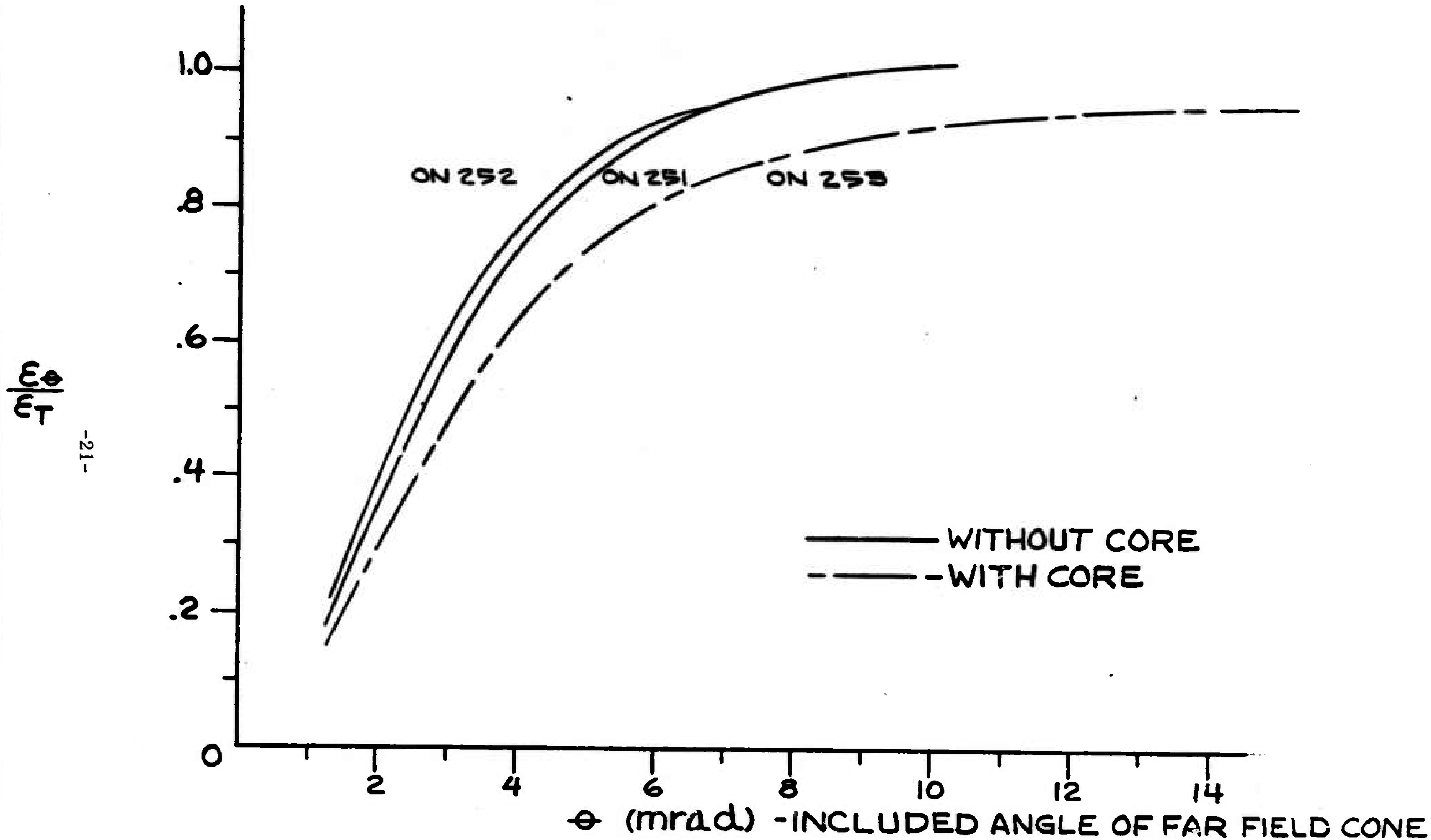

FIGURE 12 - BEAM DIVERGENCE RESULTS OBTAINED UNDER ACTIVE LASING CONDITIONS

D-2912C

III. SUMMARY OF RESULTS

Large diameter rods of good optical quality have been grown and evaluated. A limitation has been imposed on the crystal dimensions by the size of the current iridium crucible. It has been demonstrated, however, that the required diameter and length are both obtainable, and with a slightly larger size crucible but using the already developed furnace design and operating techniques a crystal could be grown from which laser rod with finished dimensions 2-inch diameter and 12 inches long, could be fabricated.

It has been possible to use a growth orientation which confines the core down the center of the cylindrical boule. Sectioning of the boules and fabrications from these sections allows the production of rods without the core; as part of this study a rod with a diameter of 3/4-inch has been fabricated and optically evaluated.

Experiments have been carried out to investigate the influence of the core on the lasing behavior of the ruby rods. A correlation has been made between the core, determined using Schlieren photography, and an uneven energy distribution in the near field lasing pattern; the core shows a general tendency to lase first as the input energy is increased. Rods fabricated without the core show a definite improvement in the energy distribution of the laser output, and in measurements of the active beam divergence.

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Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Union Carbide Corporation, Electronics Division Crystal Products R and D P. O. Box 24184, Indianapolis, Indiana 46224	2a. REPORT SECURITY CLASSIFICATION Unclassified
	2b. GROUP N/A

3. REPORT TITLE

Czochralski Ruby, Final Report

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Final Report - Period: January 1, 1967 - April 30, 1967.

5. AUTHOR(S) (Last name, first name, initial)

Keig, George A.

6. REPORT DATE February 10, 1968	7a. TOTAL NO. OF PAGES 21	7b. NO. OF REFS 4

8a. CONTRACT OR GRANT NO. Nonr-4132(00)	9a. ORIGINATOR'S REPORT NUMBER(S) SRCR-68-3
b. PROJECT NO. Task No. NR-17-710	
c. Program Code Number 3730	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
d. Authorization ARPA Order 306-62	

10. AVAILABILITY/LIMITATION NOTICES

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Office of Naval Research Department of the Navy Washington, D. C. 20360

13. ABSTRACT

The Czochralski growth technique has been developed to yield large ruby crystals which can be fabricated into rods 2 inches diameter and 12 inches long. The optical quality of the large diameter crystals and fabricated rods has been evaluated. Ruby rods of 3/4-inch diameter and having superior optical have been fabricated from sections of the large diameter boules.

Work has continued towards obtaining an improved understanding of the optical inhomogeneities in the ruby. Special emphasis has been placed on the core as this has been shown to introduce an undesirable energy distribution during lasing. Core-free rods have been fabricated from the large diameter boules and their behavior under both passive and active conditions compared to that of rods containing the core.

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